1.0

4.5
5.0
5.5

2.8

2.5

3.2

2.2

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1.1

4.0

2.0

1.8

1.25

1.4

1.6

MICROCOPY RESOLUTION TEST CHART
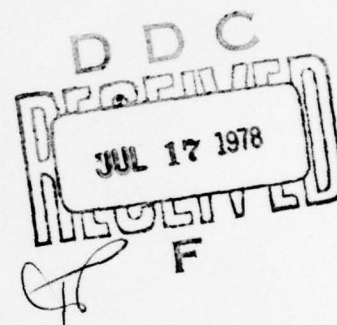NATIONAL BUREAU OF STANDARDS-1963-A

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14 NWSC/CR/RDTR-85

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DISPOSAL OF NAVY COLORED SMOKE COMPOSITIONS
AND IDENTIFICATION OF PRODUCTS OF COMBUSTION

10 Clarence W. Gilliam
Carl E. Dinerman

Naval Weapons Support Center
Applied Sciences Department
Crane, IN 47522

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31 May 1978

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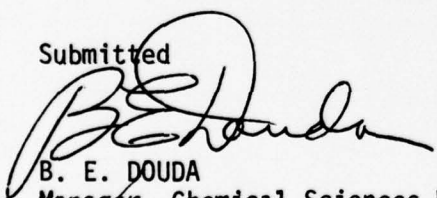Approved for Public Release; Unlimited Distribution.

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Prepared for
Commander
Naval Sea Systems Command
Washington, DC 20362

Submitted

B. E. DOUDA
Manager, Chemical Sciences Branch
Pyrotechnic Division
Applied Sciences Department

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*
An investigation was conducted to find the most environmentally acceptable and cost effective means of reclaiming or disposing of waste Navy colored smoke compositions. It is shown that the number of different kinds of excess smoke devices, the quantity related to the different kinds, cost of reclaim of each kind, and total quantity dictated that a disposal method be developed. The method of disposal chosen was incineration. A stainless steel incinerator with a 2.3 meter pathlength was constructed with a room and high temperature

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section. The room temperature section is equipped with a thermocouple to allow monitoring of the combustion process from ignition to removal of sublimed dye from the room temperature section for oxidation in the high temperature section.

All effluents were collected for gas and condensables analyses. The results were a myriad of organic compounds: aromatics, and high molecular weight alkanes. The composite was 1300 micrograms which is 65 ppm. No polynuclear aromatic compounds were found.

PREFACE

This paper was written for presentation at the Sixth
International Pyrotechnics Seminar held at Estes Park, CO,
on 17-21 July 1978.

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INTRODUCTION

This is a report on efforts sponsored by the Naval Sea Systems
Command to dispose of or reclaim the approximately 7,500 kg of excess
colored smoke compositions within the Navy system in an environmentally
permissible manner. These smoke compositions, which contain carcinogenic
or suspected carcinogenic dyes,[1-2] are contained in non-functional
pyrotechnic devices.

We now realize that the problem is much more complex than it first
appeared,[3] for reasons to be explained below, and that a more careful
analysis of the approach is needed. Having reviewed the work in detail,
we now see the need for a more complex approach.

From a chemical standpoint alone, colored smoke composition disposal
demands an extremely efficient process, with certain minimum temperature
and oxidant to fuel ratio because not only are the organic dyes suspected
to be carcinogenic, but also the products resulting from partial

[1]E. J. Owens and D. M. Ward, A Review of the Toxicology of Colored Chemi-
cal Smokes and Colored Smoke Dyes, EB-TR-74064, Edgewood Arsenal,
Aberdeen Proving Ground, MD (December 1974). Available NTIS AD-A003827.
(Available NTIS means the document may be obtained from National Technical
Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161.)

[2]M. H. Weeks and P. P. Yevich, The Toxicity of Combustion Products of
Pyrotechnics, Technical Memorandum 26-12, U.S. Army Chemical Warfare
Labs., Army Chemical Center, MD (May 1960). Available NTIS AD-474403.

[3]C. E. Dinerman and C. W. Gilliam, Ecological Disposal/Reclaim of
Navy Colored Smoke Compositions, NWSC/CR/RDTR-36, Naval Weapons
Support Center, Crane, IN (June 1976). Available NTIS AD-A030234.

oxidation/decomposition themselves can be carcinogenic as well[4]. How-
ever, in the Navy's situation, other factors are present which can
influence the ultimate choice of disposal method almost as much as
the chemistry of the smoke composition.

The Navy has many different kinds of excess smoke devices, in
greatly varying amounts, which vary in composition, candle size, types
of primer, first fire, starter and igniters (most of which contain
toxic heavy metals), types of container, whether metal, paper or
plastic. The presence of a companion flare composition in the hard-
ware presents an additional problem for any disposal method. The Mk 13
Mod 0 Marine Smoke and Illumination Signal is an example.

In the sequence of events involved in preparing a unit for disposal,
there are a number of decision points to minimize handling and preparation
costs. For instance, after the outer hardware is removed, there is left
a smoke composition enclosed in a cup or tube with primer, first fire,
etc. Different disposal methods require different preparations. For
example, there are many forms that the smoke composition can be intro-
duced into the disposal device.

The following forms assume that devices enter the disposal process
from storage, manufacturing processes, and from the array of condition
codes, and furthermore that the outer case is removed, and ignition wires
cut in every case:

---

[4]Milton L. Lee, Analysis of Polynuclear Aromatic Hydrocarbons, Indiana
University, Bloomington, IN (September 1975) pp 8-11. This Thesis can
be obtained from University of Michigan, University Microfilms, 300
North Zeeb Road, Ann Arbor, MI 48100.

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(1) Smoke composition is left in consolidated hardware, i.e., cup, tube, etc., and first fire and primer are attached.

(2) Smoke composition is left in consolidated hardware with first fire and primer removed.

(3) Smoke composition removed from consolidated hardware with first fire and primer still attached, but may be in chunk form.

(4) Smoke composition scrap from manufacturing process in chunk or fine powder.

(5) Smoke composition removed from hardware by water jet or washing from manufacturing process to give a slurry.

Preparation costs for introducing the smoke composition into a disposal device increase from form (1) to form (5). Since different disposal methods differ in the forms that they can accept, the preparation costs must be considered in the choice of a disposal method. What we wish to emphasize here is that the optimum disposal method must efficiently destroy the carcinogenic organic dyes, must not release significant amounts of toxic or carcinogenic products or particulate matter to the environment, and should require minimum preparation of the smoke composition.

The purpose of this work is to explore options and to develop alternative methods of reclamation or disposal for unserviceable Navy pyrotechnic smoke compositions so that a choice can be made as to which of these is most advantageous to the Navy.

These efforts are part of a larger plan to dispose of or reclaim chemical ingredients of non-functional colored smoke, dye marking, infrared decoy flare, and colored flare devices.

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DISCUSSION

Incineration Alone

We found it advantageous to construct a stainless steel incinerator[3]. It is 1.27 cm OD (.5 inch), 2.3 meter (90.6 inches) pathlength and will accommodate 0.6 grams of sample, which is 1.5 times larger than the sample size of previous incinerator runs.

The incinerator is designed with a room temperature section A, a high temperature, 1100K, section B, and end cap C (See Figure 1). It is operated at an oxygen flow rate of 100 cm^3/min (STP), which is turned on after the sample is ignited in the room temperature section.

The end cap of the room temperature section, see Figure 2, has an igniter wire A, a thermocouple B, which monitors the temperature of section A, after ignition, and supports the silica ignition boat C. The oxygen enters the incinerator through the one-way ball valve D. The incinerator fits into a Lindberg high temperature electric furnace.
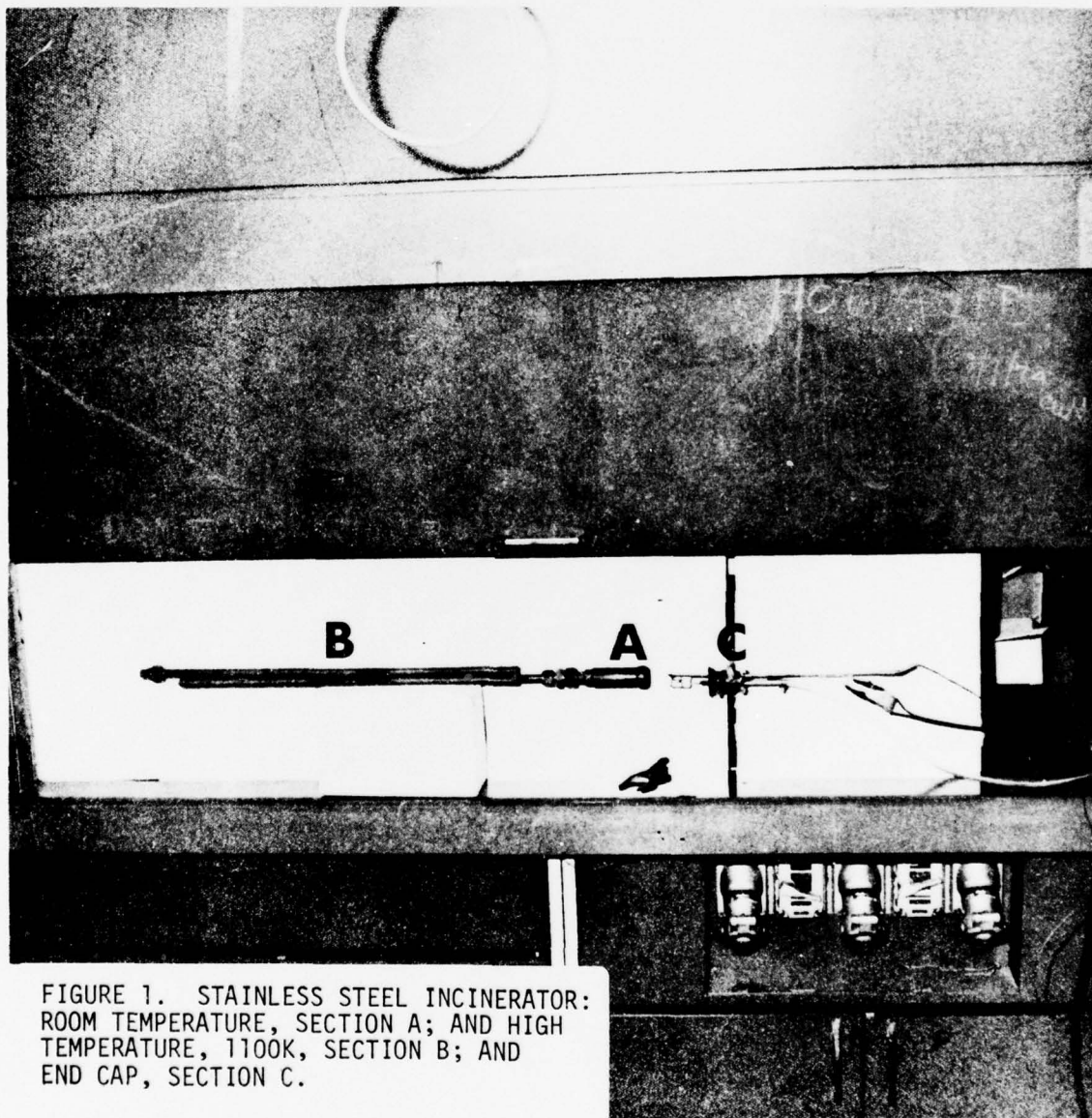
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FIGURE 1. STAINLESS STEEL INCINERATOR: ROOM TEMPERATURE, SECTION A; AND HIGH TEMPERATURE, 1100K, SECTION B; AND END CAP, SECTION C.
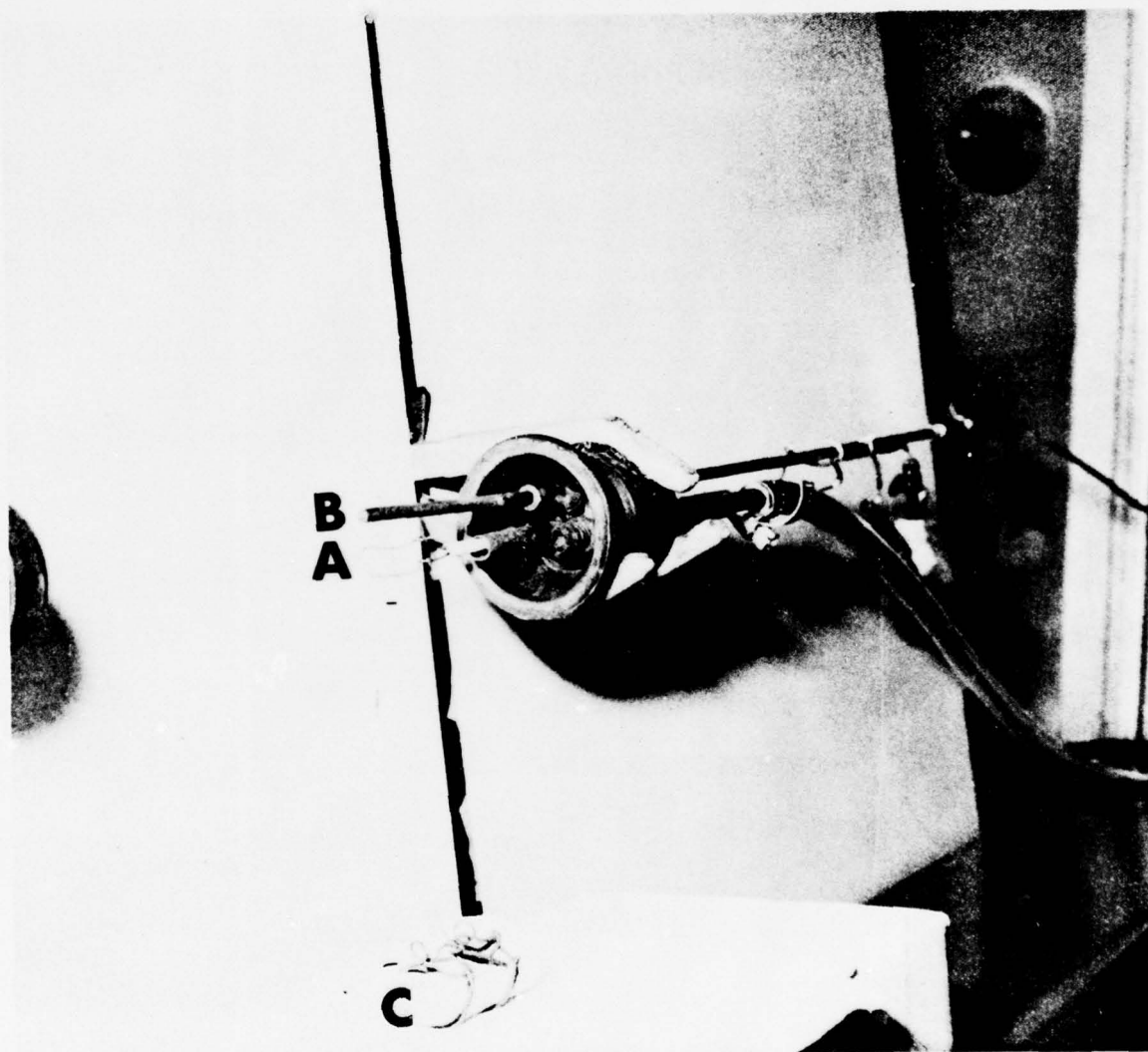
FIGURE 2. FRONT END CAP OF INCINERATOR:
A, IGNITION WIRE; B, THERMOCOUPLE AND
SUPPORT ROD FOR C, THE IGNITION BOAT;
AND D, A ONE-WAY BALL-VALVE FOR OXYGEN
SUPPLY.

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Operation of the Incinerator

Smoke composition samples are wrapped in a 6 cm^2 piece of tissue paper which serves as an ignition composition, and is placed into the silica ignition boat, A (see Figure 3) on top of the ignition wire. The electrodes, B, are fastened to clip leads, C, and the end cap is securely fastened to the incinerator with a ball joint clamp, D, and the oxygen is supplied to the system through the transmission tube, E.

The complete assembly, Figure 4, shows A, the electric furnace; B, the high temperature section which is held isothermally at 1100K; C, the room temperature portion; D, the end cap assembly; E, the all glass cold trap; F, the gas collection bag; and G, the furnace controls.

Prior to ignition, the oxygen supply is turned off. At ignition, $KClO_3$/sugar fuel sublimes the dye, most of which collects on the walls of the room temperature section of the incinerator. The ignition is visually detected by the stream of particulates observed in the cold trap carried by large amounts of gaseous (CO_2, H_2O vapor) products from this ignition, which in the small volume of the incinerator, causes a momentary pressure buildup. This gas pressure forces some dye through the room temperature and heated portions of the incinerator, where the short residence time and the lack of pure oxygen (recall that the oxygen flow isn't turned on until a few seconds after ignition) causes particulates to form.

The oxygen flow is resumed, and the room temperature section is brought to 770K quickly, which is well above the sublimation temperature, 550K, of the dyes.

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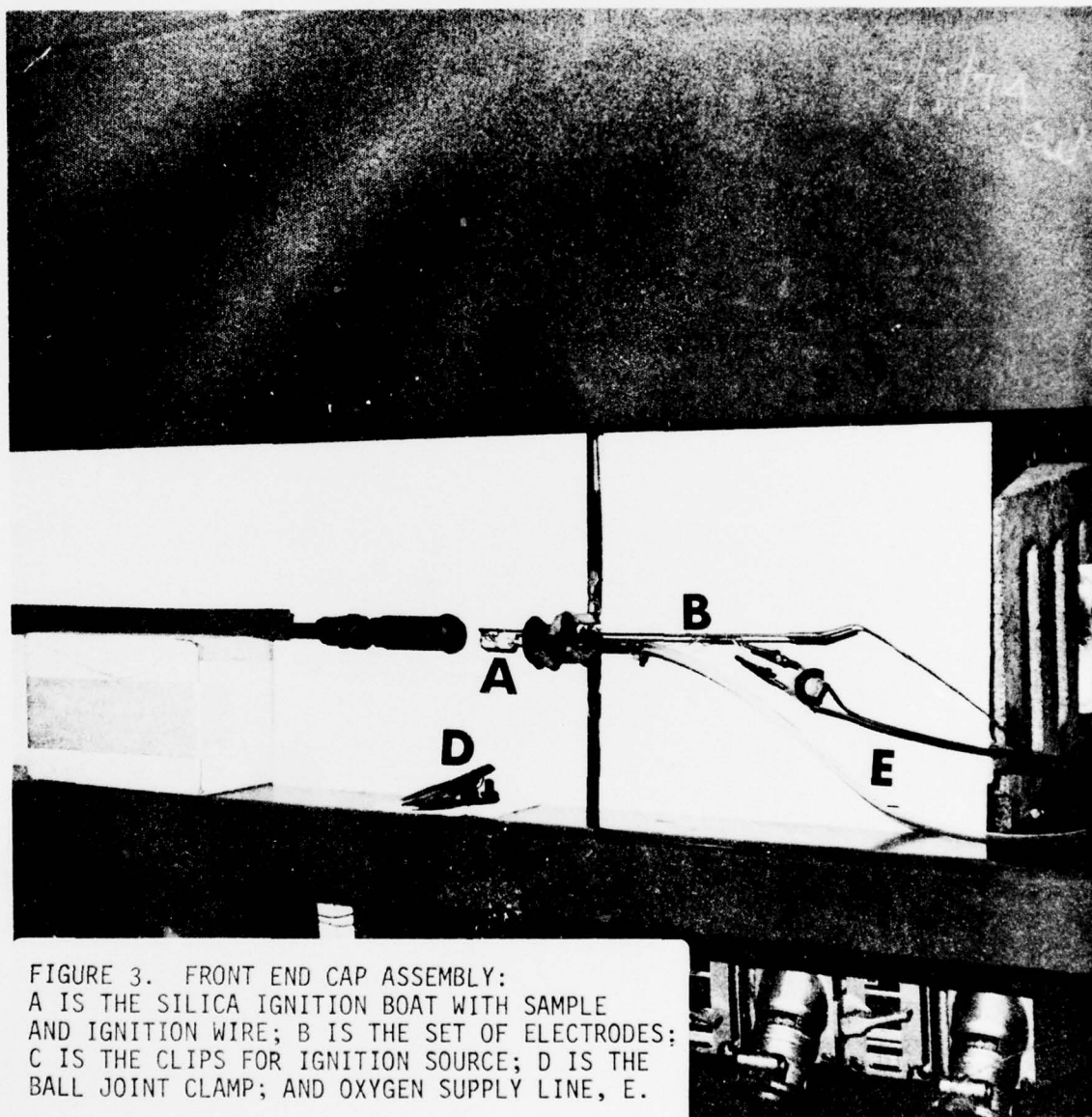
FIGURE 3. FRONT END CAP ASSEMBLY:
A IS THE SILICA IGNITION BOAT WITH SAMPLE
AND IGNITION WIRE; B IS THE SET OF ELECTRODES;
C IS THE CLIPS FOR IGNITION SOURCE; D IS THE
BALL JOINT CLAMP; AND OXYGEN SUPPLY LINE, E.

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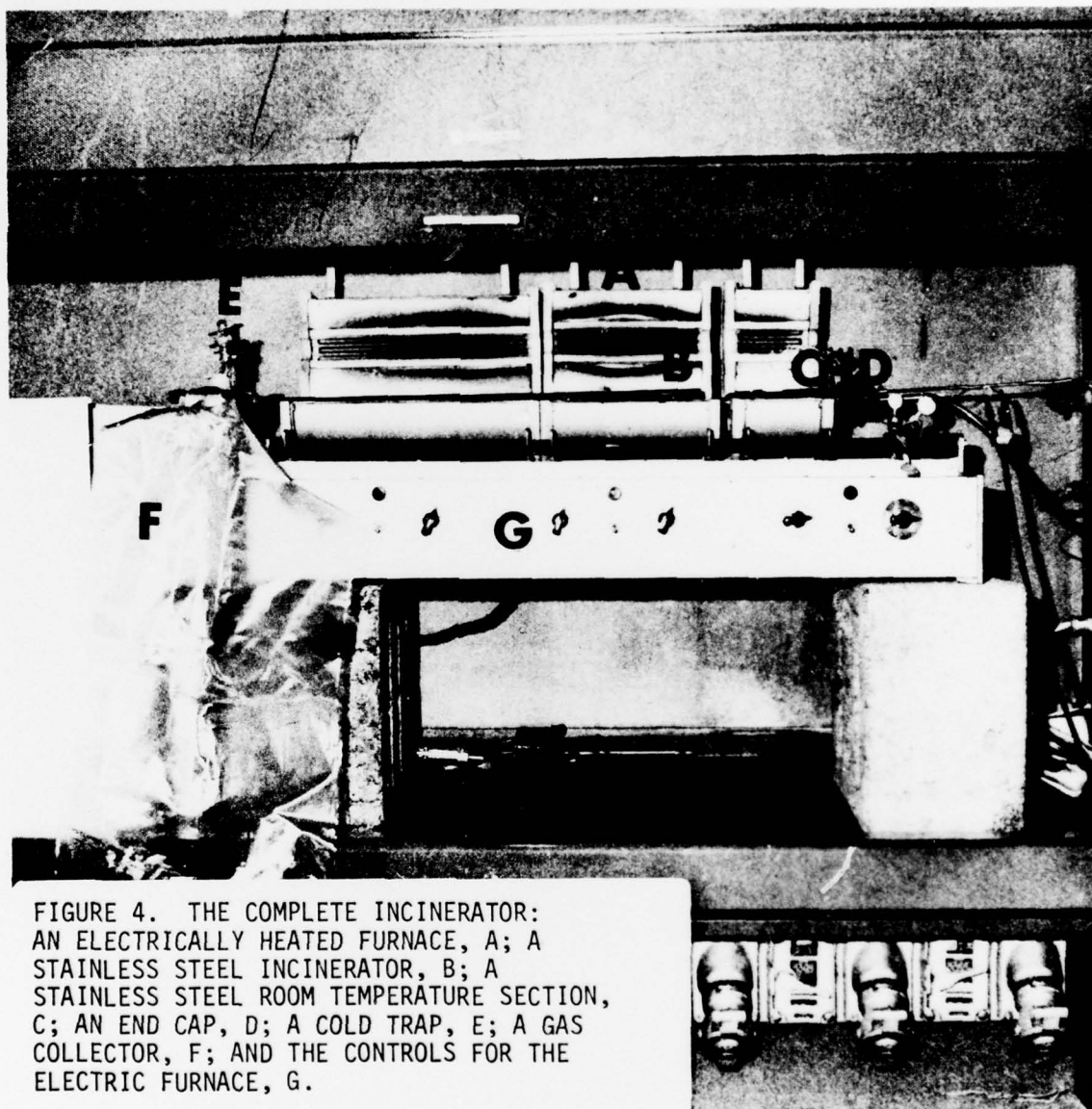
FIGURE 4. THE COMPLETE INCINERATOR: AN ELECTRICALLY HEATED FURNACE, A; A STAINLESS STEEL INCINERATOR, B; A STAINLESS STEEL ROOM TEMPERATURE SECTION, C; AN END CAP, D; A COLD TRAP, E; A GAS COLLECTOR, F; AND THE CONTROLS FOR THE ELECTRIC FURNACE, G.

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Point A, Figure 5, is the first exotherm concurrent with the resumption of oxygen flow, B is its maximum; at point C, the exotherm ends. The sublimed dye which coated the walls of the room temperature section at ignition now begins to resublime causing a second exotherm between points D and E.

The residence time of seven seconds for the process was calculated from the flow rate, temperature, incinerator pathlength and tube's inside diameter.

The isothermal portion between E and F is 770K and remains there for 11 minutes; the total reaction time is 33 minutes.

Sample Preparation

At the end of the run, the contents of the gas bag are allowed to expand into an evacuated 10 cm pathlength infrared gas cell for infrared spectral analysis.

The cold trap is disconnected, rinsed thoroughly (five times with 10 cc of glass distilled benzene) to remove benzene solubles and particulates. Each washing is poured into the soxhlet extractor through the thimble located in the extraction tube. Condensables and particulates are extracted overnight. The final volume is reduced to 10 cm^3 for GC/MS analyses.

Pilot Plant Design

The point to be made is that, in a pilot plant, the incinerator volume must be large enough to avoid pressure buildups which cause surges in the combustion chamber with subsequent reductions in effluent residence time. It is not enough to merely design the
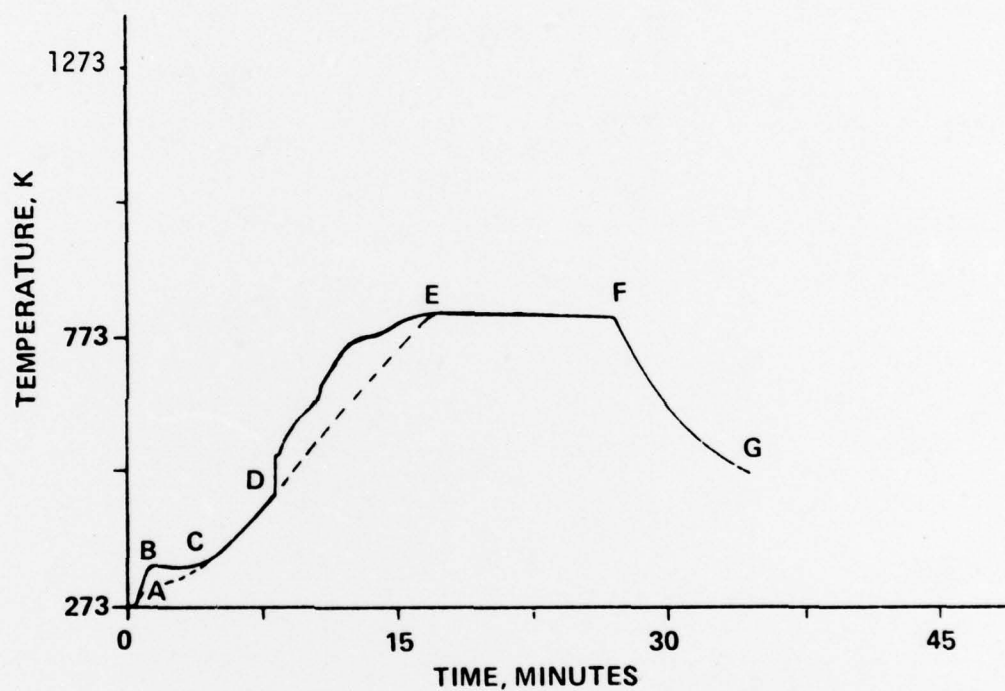
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FIGURE 5. TIME/TEMPERATURE PROFILE OF FIFTIETH
MK 13 MOD 0 INCINERATOR RUN: SAMPLE
WEIGHT, 0.58G, RESIDUE, 0.099G, AND DOT-
TED LINE IS THE BACKGROUND RUN WITH
THE TISSUE PAPER IGNITER. ALL OTHER
CONDITIONS ARE THE SAME AS EXPERI-
MENTAL.

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incinerator walls to withstand the pressure; the pressure must be avoided in the first place.

Another factor to be considered in pilot plant design is the particulate size in the incinerator effluent. The particle size distribution can influence the type of particulate collector used such as cyclone, wet scrubber, electrostatic precipitator or fabric filter.

Using a microscope, we made rough examination of the particulates from two recent Mk 13 runs. In both cases, we found a large number of the particles smaller than 10 μm, with occasional particles with sizes ranging from 10 to 50 μm. These particles were collected in the incinerator effluent trap, rinsed with benzene to collect PAH's, and subsequently allowed to dry. It's possible that the particles, at the time they appear in the effluent trap, are agglomerated to some degree because of the water present as a combustion product. If this is the case, then observing the particles after they have dried, with possible deagglomerization, may be a "worst case;" that is, the population less than 10 μm may be greater than the population observed.

These measurements of particle size will have meaning in a pilot plant design only if it is determined that an incinerator alone will be used, and not some combination such as incinerator/fluidized bed as an example.

Source Emission Standards for PAH

The Indiana Air Pollution Control Board was contacted, and it was determined that there are no PAH source emission standards for Indiana. However, it was determined that suspended particulates, carbon monoxide,

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total volatile hydrocarbon are regulated by APC 15[12] of 27 January 1972. Also, regulation APC 7 of 6 December 1968[12] states that, "All new incinerators shall be multiple chamber or equivalent thereof".

The Environmental Protection Agency, Strategies and Standards Division, Research Triangle Park, NC was contacted. We determined, from this contact, that there are no existing nation-wide PAH source emission standards. However, work is being done to set source emission standards for coke oven effluents, with the main thrust directed toward benzene solubles. This standard will include PAH's and aza-arenes.

However, a Threshold Limit Value[13] (average concentration for normal eight-hour workday or 40-hour work week, to which nearly all workers may be repeatedly exposed, day after day, without adverse effect) of .2 mg/m^3 has been established for particulate PAH's (see benzene solubles). *This value, though, cannot be directly converted* to a source emission standard, which is what is needed for the present project.

Since there are no existing source emission standards and most likely won't be for at least one or two years, we have no choice but to rely on existing standards; for example, those mentioned above. However, it should be kept in mind that in two years this incinerator will come on stream and new standards will apply.

─────────────

[12]Air Pollution Control Board, 1330 West Michigan Street, Indianapolis, IN 46206.

[13]TLVS® Threshold Limit Values for Chemical Substances in Workroom Air Adopted by American Conference of Governmental Industrial Hygienists for 1976. (Available from Secretary-Treasurer, P. O. Box 1937, Cincinnati, OH 45201.)

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Analytical Results and Discussion

We negotiated a contract with Indiana University, Chemistry
Department, with Dr. Milos Novotny to qualify, identify and quantitate
the compounds in the effluent stream. Dr. Novotny and graduate
assistant, Mr. James Jorgenson reported as follows:

By means of gas chromatographic and mass spectroscopic techniques,
representative samples of combustion products of materials containing
the following chemical compounds were analyzed:
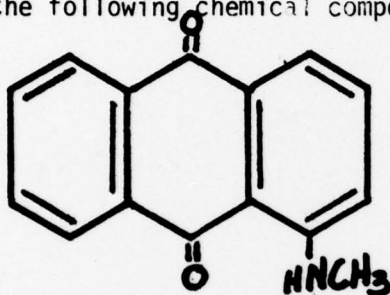
1-Methylamino-Anthraquinone
(MW 237)

and
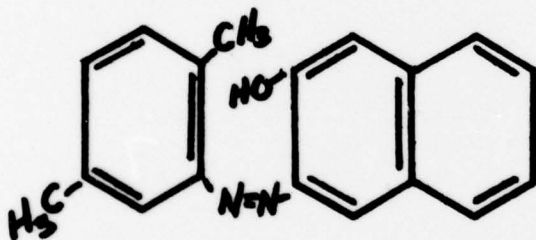
Xylene-azo-β-Naphthol
(MW 276)

These samples were analyzed in support of the Pyrotechnic Disposal
Program at the Naval Weapons Support Center, Crane, Indiana.

For initial screening of samples, high-performance (capillary) gas
chromatography with the flame ionization detection was used. Glass
capillary column (20 meters x 0.28 mm. i.d.) coated with SE-52 silicone
elastomer was used in the Perkin-Elmer 3920 gas chromatograph (program-
ing from 70 to 240°C). A number of chromatographic peaks appears in the

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studied range, but the lower portion up to peak no. 16 (see Figure 6)
is inconsistent for different samples. The peaks appearing in the
upper portion of gas chromatogram (Figure 6) (16-50) have been studied
by means of combined capillary gas chromatography/mass spectrometry
(Hewlett-Packard dodecapole 5980 A instrument). The total-ion-current
chromatogram is shown in Figure 7.

Since the initial compounds contained nitrogen, additional recordings
were obtained with the thermionic (nitrogen-sensitive detector) (see
Figure 8). Total of 42 mass spectra was recorded. The results of
analyses are summarized in Table I, which also shows approximate amounts
of the analyzed compounds.

Important conclusions of the analyses are:

(1) Many of the detected compounds are alkanes, which are not uncommon
in various combustion mixtures. Several of the detected compounds are
presently of unknown structures, since mass spectrometry alone cannot
provide the answer (IR- and NMR- spectroscopy could probably provide
their structures) due to absence of such mass spectra in the literature
and mass-spectral catalogues. Ab initio interpretation was not feasible.

(2) Approximately eight studied substances appear to contain nitrogen
(odd mass numbers). Since they do not yield response in the thermionic
detector, it is likely that the compounds have nitro groups in their
molecules.

(3) No polynuclear aromatic hydrocarbons were detected in these
samples.

18
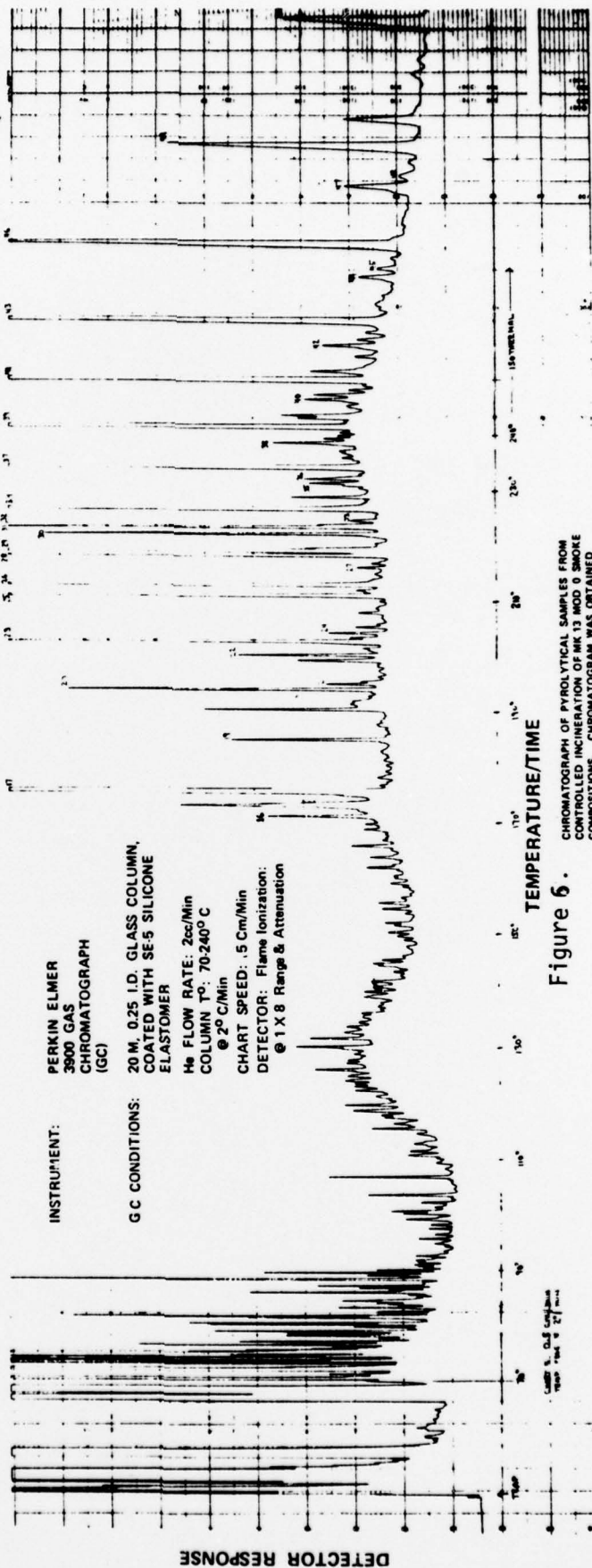
INSTRUMENT: PERKIN ELMER 3900 GAS CHROMATOGRAPH (GC)

G C CONDITIONS: 20 M. 0.25 I.D. GLASS COLUMN, COATED WITH SE-5 SILICONE ELASTOMER

He FLOW RATE: 2cc/Min
COLUMN T°: 70-240°C @ 2°C/Min

CHART SPEED: .5 Cm/Min

DETECTOR: Flame Ionization: @ 1 X 8 Range & Attenuation

DETECTOR RESPONSE

TEMPERATURE/TIME

Figure 6. CHROMATOGRAPH OF PYROLYTICAL SAMPLES FROM CONTROLLED INCINERATION OF MK 13 MOD 0 SMOKE COMPOSITIONS. CHROMATOGRAM WAS OBTAINED WITH A GLASS CAPILLARY COLUMN, 20 METERS X 0.25 MILLIMETERS, I.D., COATED WITH SE-52 SILICONE ELASTOMER WITH A FLAME IONIZATION DETECTOR PROGRAMMED FROM 70-240°C AT 2° C/MIN.

ATTENUATION 1 X 8

19
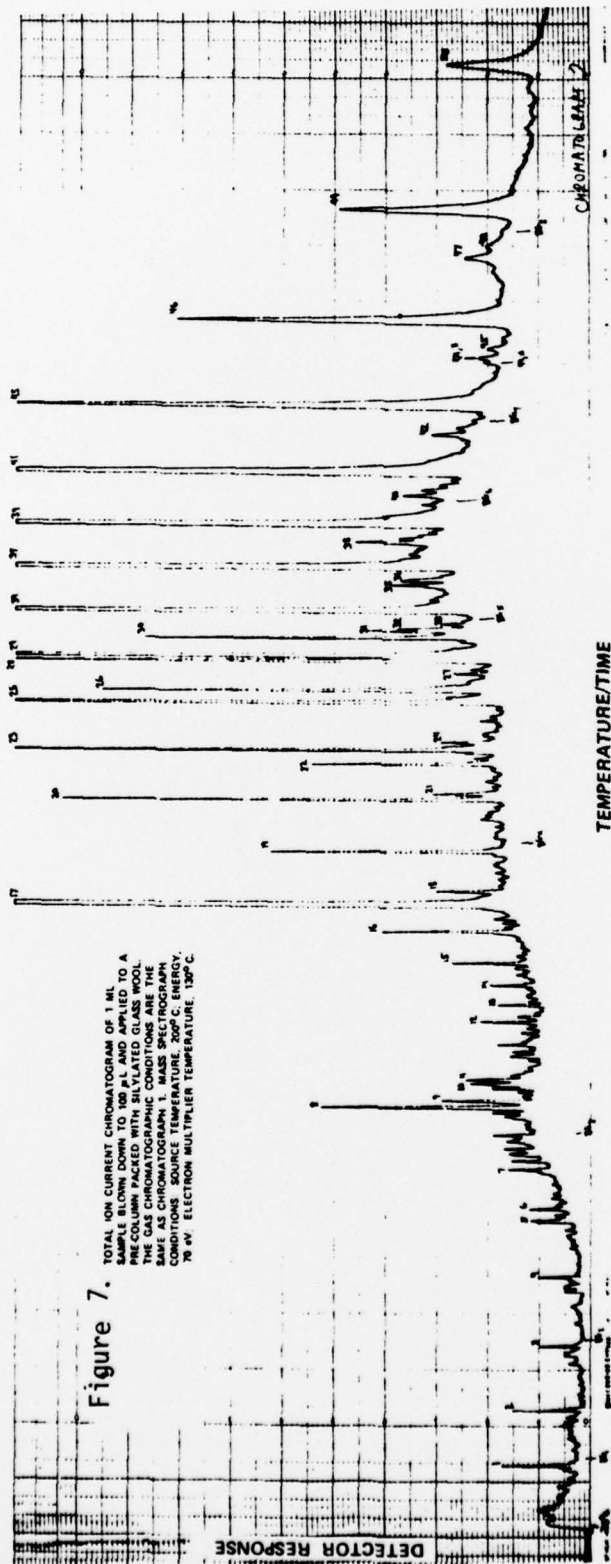
Figure 7. TOTAL ION CURRENT CHROMATOGRAM OF 1 ML SAMPLE BLOWN DOWN TO 100 µL AND APPLIED TO A PRE-COLUMN PACKED WITH SILYLATED GLASS WOOL. THE GAS CHROMATOGRAPHIC CONDITIONS ARE THE SAME AS CHROMATOGRAM 1. MASS SPECTROGRAPH CONDITIONS: SOURCE TEMPERATURE, 200°C. ENERGY, 70 eV. ELECTRON MULTIPLIER TEMPERATURE, 130°C.
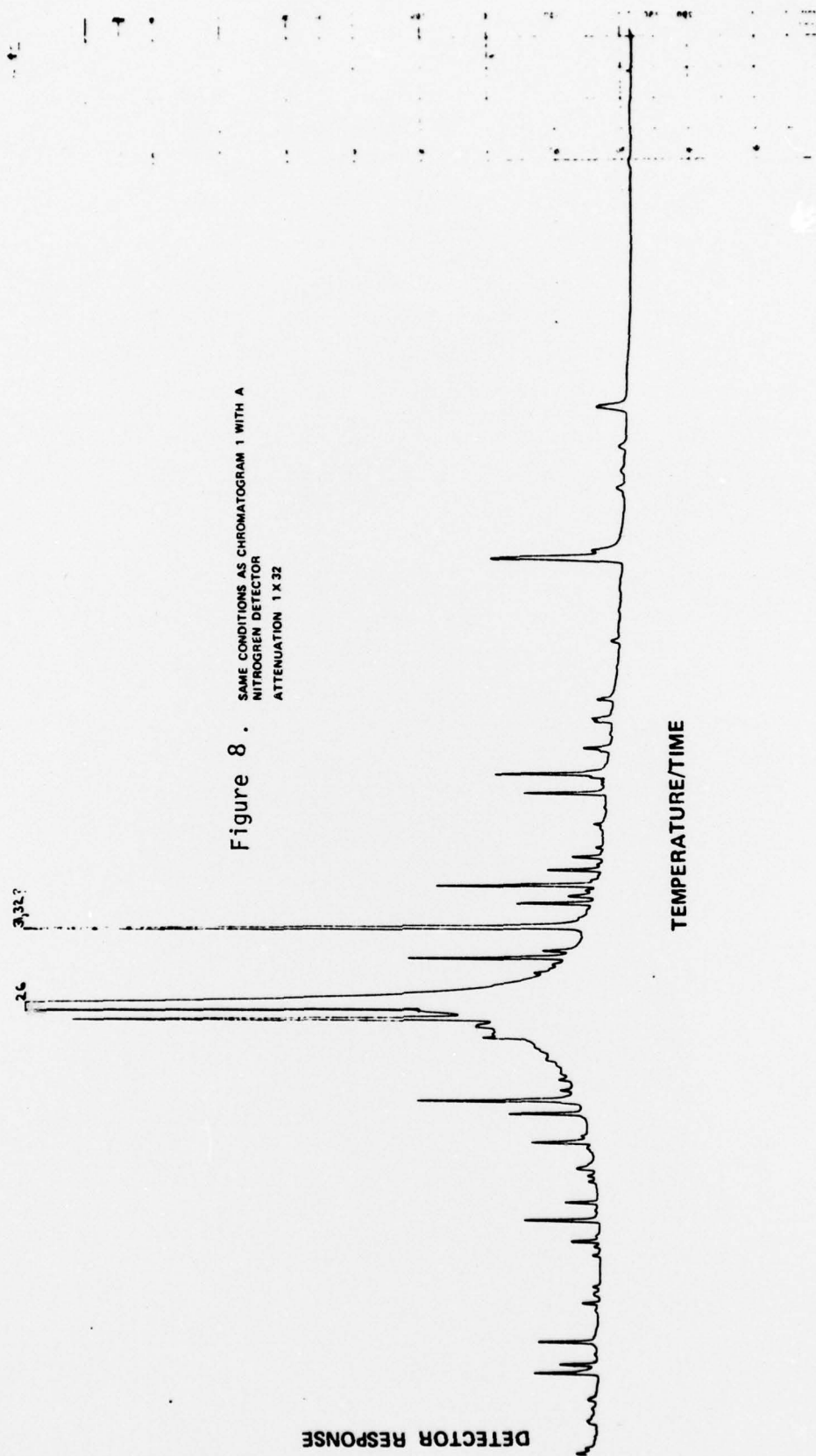
Figure 8. SAME CONDITIONS AS CHROMATOGRAM 1 WITH A
NITROGREN DETECTOR
ATTENUATION 1 X 32

TEMPERATURE/TIME

DETECTOR RESPONSE

Table I: Analysis of Soxhlet Extraction of Effluents from Laboratory Incinerator (2.3 meters long at 100 cm³/min of O_2 at STP), Mk 13 Mod 0, .58 g sample.

Compound Number (Peak No.)	Total Wt. in Nanograms	Sample ppm	Molecular Weight	Comments
16	70	0.33	223	Aromatic
17	2,000	9.5	282	$C_{20}H_{42}$
18	40	0.19	-	No spectrum
19	100	0.48	296	$C_{21}H_{44}$
20	150	0.71	310	$C_{22}H_{46}$
21	50	0.24	-	Too weak
22	100	0.48	329	Aromatic
23	500	2.4	324	$C_{23}H_{48}$
24	40	0.19	314	Aromatic
25	1,000	4.8	338	$C_{24}H_{50}$
26	200	0.95	299	Definitely contains nitrogen (possibly <u>nitro</u> compound)
27	30	0.14	-	Too weak
28	500	2.4	249	Aromatic (related to no. 30)
29	1,000	4.8	352	$C_{25}H_{52}$
30	200	0.95	279	Aromatic (related to no. 28)
31	50	0.24	326	Aromatic
32	40	0.19	323	Weak
33	10	0.05	-	Too weak
34	1,000	4.8	366	$C_{26}H_{54}$
35	30	0.14	-	Too weak
36	30	0.14	261	Weak
37	1,000	4.8	380	$C_{27}H_{54}$
38	40	0.19	351	Weak
39	1,000	4.8	394	$C_{28}H_{58}$
40	20	0.10	-	Too weak
41	1,000	4.8	408	$C_{29}H_{60}$
42	30	0.14	-	Too weak
43	1,000	4.8	422	$C_{30}H_{62}$
44	40	0.19	-	Too weak
45	30	0.14	-	Too weak
46	1,000	4.8	436	$C_{31}H_{64}$
47	100	0.48	-	Too weak
48	30	0.14	-	No spectrum
49	500	2.4	450	$C_{32}H_{66}$
50	500	2.4	464	$C_{33}H_{68}$

(4) Since residues of samples were observed at the column inlet, the presence of heavy (non-volatile) compounds is clearly suggested. Gas chromatography is here a limited method, and liquid-chromatographic investigations would be needed to study such non-volatile components.

(5) Although none of the original dye compounds were detected, their thermal stability may be limited under gas-chromatographic conditions. Thus, some of the nitrogen-containing compounds as detected from the mass spectra (Figure 8) may actually be artifacts of the original composition.

What we learned:

(1) That an incinerator of sufficient size and volume will eliminate pressure pulses caused by rapid burning of organic materials upon ignition and onset of oxidant flow.

(2) That increased turbulence with the same residence time and oxidant/fuel ratio should give satisfactory results.

(3) That the incinerator should follow the basic two-zone design; i.e., a room temperature zone first followed by a high temperature zone to complete the oxidation process.

(4) That a decrease in sample weight:

    a. Aids burning efficiency because pressure pulses are alleviated,
    b. Aids residence time because pressure pulses are alleviated, and
    c. Prolongs the time to complete the disposal operation, but does not impose prohibitive costs.

(5) That most of the particulates, when formed, are less than 10 μm. The size of particle that we observed necessarily dictates a sophisticated scrubbing system to meet clean air standards (State Regulations).

(6) That the nature of the reaction products is related to oxidation efficiency.

(7) That complex dye structures may give undesirable reaction products if incinerator's parameters (time, temperature, and pressure) are not re-optimized; i.e., smaller sample size and longer residence time are examples.

(8) That complete definition of reaction products requires an array of high performance analytical instruments.

(9) We found no polynuclear aromatic compounds for all Mk 13 Mod 0 runs.

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